

Emissions of volatile organic compounds from heated needles and twigs of *Pinus pumila*

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Abstract: A study was conducted to explore the mechanism that emissions of volatile organic compounds (VOC) from heated needles and twigs (200°C, within 15 min) of *Pinus pumila* affect fire behaviours using the technology of Thermal Desorption - Gas Chromatography - Mass Spectrometry (TD-GC-MS). The results indicated that the main components of VOC from heated needles and twigs are terpenoids. Most of these terpenoids are monoterpenes. Terpenoids account for 72.93% for the needles and 92.40% for the twigs of the total VOC, and their emission ratios are 61.200 $\mu\text{g}\cdot\text{g}^{-1}$ and 217.060 $\mu\text{g}\cdot\text{g}^{-1}$ respectively. Heated twigs can emit more terpenoids than heated needles because twigs had more volatile oils than needles. In actual fires, these large amounts of terpenoid emissions, especially the monoterpene emissions, have strong effects on fire behaviors that are not only in the initial stage but also in the fast propagation stage of fires. These flammable gases are capable of causing violent combustion and creating crown fires. In addition, if these gases accumulate in an uneven geographical area, there will be a possible for eruptive fires and/or fires flashover to occur.

Keywords: forest fire; *Pinus pumila*; volatile oil; volatile organic compounds (VOC); terpenoids

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Introduction

Forest fires often result in significant property damage and casualties. The content of volatile oils in plants has important influences on the flammability and combustibility of fuels. Volatile oils may increase the combustibility of plant material by increasing the probability of ignition (White 1994; Zedler 1995). The main components of volatile oils are terpenoids, such as monoterpenes, oxygen-monoterpenes and sesquiterpenes. Volatile oils are mainly in leaves and small twigs of plants. The coniferous species have higher content of volatile oils than the broad-leaves species. The volatile oils of coniferous needles are about 2% of plant tissue. Moreover in spruce, fir and some pine species, the volatile oils reached 6% (Wen et al. 1999). The calorific value of volatile oils is 32.36 $\text{MJ}\cdot\text{kg}^{-1}$, which is twice as much as (16.127 $\text{MJ}\cdot\text{kg}^{-1}$) that of cellulose and hemicellulose. Owens et al. (1998) reported that the higher concentration of limonene (one kind of monoterpenes) is positively related to plant flammability and could increase flammability by 30% over the range of concentrations found in *Juniperus ashei*.

Researchers have paid lots of concerns to the influences of volatile oils on forest fires. Volatile oil content has been an important index to evaluate the flammability of different tree species (Pompe and Vines 1966; Shu et al. 1999; Chen et al. 1995). Ormeño et al. (2009) reported that monoterpene concentration in leaf litter is positively correlated to flame height and negatively correlated to both flame residence time and ignition delay. Many studies assumed that volatile oils of plant favor fire due to their enormous flammability. However, up to now, little is known about the mechanism how volatile oils affect fire behaviours. Pompe and Vines (1966) found that though the calorific values of volatile oils are very high, and they have no prominent contribution to the total calories from fuel combustion due to small amount. Their contribution is shown at the initial stage of fires, especially when the fuels still have some water. Fan (1993) reported that the forests composed of species with high volatile oil content are prone to present high-intensity fire behaviours. Most

recently, the study on terpenoid emissions from heated vegetation has provided a good method to explore the mechanism how volatile oils affect fire behaviours (Chetehouna et al. 2009). As the boiling points of main volatile oils are very low (Table 1), a large amount of terpenoids will be emitted from plants under heated conditions, i.e., before the pyrolysis of plant tissue (Granström 2003; Maleknia et al. 2009a, 2009b). The large amount and very flammable gases would have strong effects on fire propagation.

Table 1. Formula, molecular weight and boiling point of the main terpenoids discussed in present paper*

Compounds	Formula	Molecular weight	Boiling point (°C)
santene	C ₉ H ₁₄	122	140-141
fenchene	C ₁₀ H ₁₆	136	142-143
tricyclene	C ₁₀ H ₁₆	136	152-153
α -pinene	C ₁₀ H ₁₆	136	155-156
camphene	C ₁₀ H ₁₆	136	159-160
sabinene	C ₁₀ H ₁₆	136	163-164
β -pinene	C ₁₀ H ₁₆	136	165-167
α -terpinene	C ₁₀ H ₁₆	136	173-175
trans- β -ocimene	C ₁₀ H ₁₆	136	174-175
eucalyptol	C ₁₀ H ₁₈ O	154	174-177
D-limonene	C ₁₀ H ₁₆	136	176-177
β -phellandrene	C ₁₀ H ₁₆	136	178-179
γ -terpinene	C ₁₀ H ₁₆	136	181-183
terpinolene	C ₁₀ H ₁₆	136	183-185
neo-allo-ocimene	C ₁₀ H ₁₆	136	188-189
2(10)-pinen-3-one	C ₁₀ H ₁₄ O	150	217-218
myrtenal	C ₁₀ H ₁₄ O	150	220-221
verbenone	C ₁₀ H ₁₄ O	150	227-228
sesquiterpenes	C ₁₅ H ₂₄	204	250-280

Note: *Data are from (<http://webbook.nist.gov/chemistry/cas-ser.html>).

P. pumila is an evergreen shrub at height of 3–6 m, and its trunks creep along the ground usually 10 m long or more. Distributing on higher altitude, generally above 800 m (Jiang 1982), *P. pumila* has cold tolerance and resistance to strong winds, and can adapt to the infertile soil with rocks (Xu 1998). In the coniferous forests in northern areas of China, *P. pumila* forest is one of the most abundant bio-diversity forest types (Okitsu 1998) and plays an important role on conserving soil and water (Okuda 2008). However, the main disturbance factors are fires for *P. pumila* forest (Shu et al. 2003) because *P. pumila* is very flammable due to having large amounts of volatile oils in its body (Shan et al. 2003). In 2001 and 2002, serious large fires occurred in the *P. pumila* forests (Du and Wang 2007). The fires in *P. pumila* forests always have characteristics with violent combustion, heavy smoke, high fire intensity and fast fire spreading speed. In addition, *P. pumila* is prone to crown fires. In the present paper, the VOC emissions from heated needles and twigs (200°C) of *P. pumila* are studied for exploring the mechanism how volatile oils affect fire behaviours.

Materials and methods

Sample collection of needle and twig

Needle and twig samples were collected in late July of 2009 in the north-western of Heilongjiang Province at Huzhong Forest Bureau (E 122°37'–124°16', N 51°14'–52°25'). The samples collected from healthy and vigorous plants were placed in ventilated, dry places in laboratory. The air-dry weight of samples was determined until the weight was constant about 1 month later.

VOC collection from heated needle or twig samples

The methods for sampling VOC emissions from heated samples are illustrated in Fig. 1. Vacuum oven (Volume: 50 L) was used to ensure that the VOC emissions from heated materials were not lost. The inlet and outlet of the vacuum oven were connected with Teflon tubes. A glass tube (15.0 cm \times 0.3 cm; Chrompack, Middelburg, the Netherlands) containing Tenax-TA (60 to 80 mesh, Chrompack) was the volatile trap. A portable air sampler (QC-1; Beijing Municipal Institute of Labor Protection, China) served as a pump. All parts were connected by Teflon tubes. First, the vacuum oven was heated until the temperature was up to 200°C. Then the air-dry needles or twigs (about 15 g) were put into the oven. After 5 min, the sampling process started. Sampling time was 10 min and the flow rate of pump was 0.1 L·min⁻¹. Sampling processes were repeated three times, and then the mean value and standard error were calculated. After the sampling had been finished, the glass tubes with the adsorbed volatiles were sealed and placed in a desiccator.

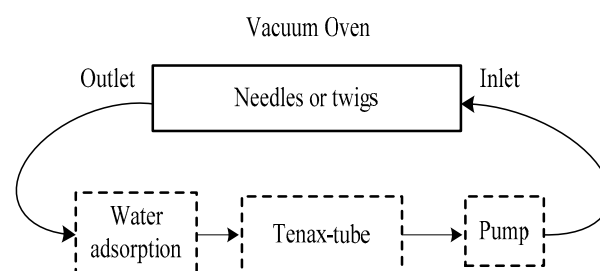


Fig. 1 Sampling of VOC emissions from heated needles or twigs of *P. pumila*.

VOC analyse methods

The collected VOC were detected with Gas Chromatography - Mass Spectrometry (GC - MS) (Agilent 6890N-5973N GC-MSD, US). Volatiles were released from the absorbent by heating the trap in a CDS 8000 (Sample concentrator, US) at 200°C for 10 min, and a carrier gas (Helium) flushed the sample toward a cold trap at 30°C. By heating the cold trap to 250°C, the volatiles were transferred into the analytical column (DB-624 capillary column, 30 m \times 0.25 mm \times 1.4 μ m). The column was programmed from 30°C to 230°C, with the Helium pressure of 6 psi.

The MS was operated in the 70eV EI ionization mode. Scanning was done from 29–500 amu.

The identification of individual components was based on a comparison of calculated retention indices on apolar column with literature data (NIST WebBook 2005). The quantification of the different terpenoids was presented in α -pinene equivalent. The quantification of α -pinene was carried out by direct liquid injection in the Tenax TA tubes and had been calibrated in mass range 1.3 ng–13 μ g.

According to formula (1) to calculate the emission ratios of different terpenoids from heated samples per unit mass within 15 min:

$$R = \frac{M_1 \times V}{R_1 \times t \times M} \quad (1)$$

where, R is the emission ratio of different terpenoids from heated samples per unit of mass within 15 min ($\mu\text{g}\cdot\text{g}^{-1}$), M_1 is the amount of different terpenoids in glass tube (μg), V is the volume of vacuum oven (50 L), R_1 is the air flow rate of pump (0.1 $\text{L}\cdot\text{min}^{-1}$), t is the sampling time (10 min), and M is the air-dry weight mass of samples (g).

Results and discussion

Terpenoid emissions are in close relation with temperature (Maleknia et al. 2009b). The terpenoid emissions increase with an increase in temperature, until temperature reaches a threshold. When the temperature is over the threshold, the terpenoid emissions will decrease (Chetehouna et al. 2009). Therefore, the temperature plays a key role in terpenoid emissions. In Table 1, it is shown that the boiling points of main components of volatile oils are below 200 °C. So in this study, 200 °C was designed as the heated temperature according to many other previous studies (Maleknia et al. 2009); Granström 2003; Maleknia et al. 2009a; Chetehouna et al. 2009).

VOC emissions from heated needles of *P. pumila*

The 41 compounds were identified in VOC emissions from heated needles of *P. pumila*, most of which were terpenoids, accounting for 72.93% of the total VOC. (Fig. 2). The large amounts of compounds were shown in Fig. 3. The small amounts of other compounds were: (1) iso-butyronitrile, (2) acetone, (3) 2-methyl-furan, (4) trichloromethane, (5) acetic acid trichloro-ethyl ester, (6) benzene, (7) acetic acid, (8) 3-methyl-butanal, (9) 2-ethoxy-ethanol, (10) dimethyl-disulfide, (11) ethylbenzene, (12) β -ocimene, (13) 1-methyl-3-(1-methylethyl)-benzene, (14) eucalyptol, (15) 2-ethyl-1-hexanol, (16) 3-methyl-2(5H)-Furanone, (17) 3,6-dimethoxy-9-(2-phenylethynyl)-fluoren-9-ol, (18) (2-methyl-1-propenyl)-benzene, (19) 4-(4-Chlorophenyl)-2,6-diphenylpyridine, (20) acetophenone, (21) 1-tridecene, (22) (Z)-2-dodecene, (23) 6,6-dimethyl-2-methylene-bicyclo[2.2.1]heptan-3-one, (24) naphthalene.

The 10 terpenoids were identified in Table 2. The dominating monoterpenes ($(\text{C}_5\text{H}_8)_2$) were α -pinene, camphene, β -pinene, D-limonene, 3-carene, neo-allo-ocimene, and santene, accounting for 59.60%, 14.25%, 11.93%, 5.63%, 2.88%, 2.02%, and 1.63% of the total terpenoids, respectively (Table 2). Similar results were reported in VOC emitted from the heated *Rosmarinus officinalis* plants (Chetehouna et al. 2009), the drying sawdust of Norway spruce (*Picea abies*) (Granström 2003), and eucalypt at different high temperatures (Maleknia et al. 2009a, 2009b). The components of terpenoid VOC are not the same with the compositions of volatile oils, that is to say, there are large amount of sesquiterpenes ($(\text{C}_5\text{H}_8)_3$) and oxygen-monoterpenes in volatile oils (Jin et al. 1994). But there no sesquiterpenes and few oxygen-monoterpene were detected in present results. The reason may be that the boiling points of sesquiterpenes and oxygen-monoterpenes are higher than that of monoterpenes (Table 1).

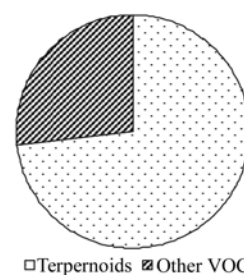


Fig. 2 Terpenoids and other VOC in the total emissions from heated needles (200 °C, 15min) of *P. pumila* in mid-Sep., 2009

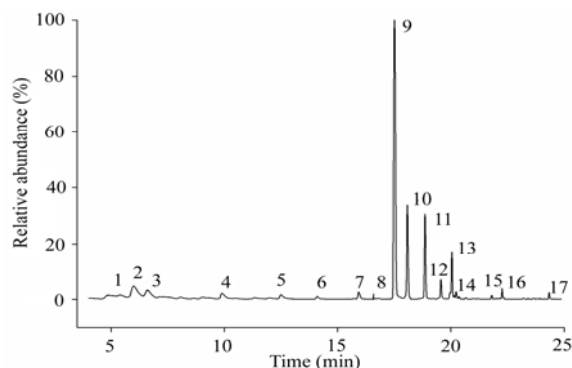


Fig. 3 Chromatographic profiles of VOC emissions from heated needles (200 °C, 15min) of *P. pumila* in mid-Sep., 2009.

(1) pentane; (2) methylene chloride; (3) hexane; (4) 1-methoxy-2-propanol; (5) toluene; (6) hexanal; (7) santene; (8) styrene; (9) α -pinene; (10) samphene; (11) β -pinene; (12) 3-carene; (13) limonene; (14) β -phellandrene; (15) nonanal; (16) neo-allo-ocimene; (17) decanal.

There are also other compounds in the VOC emissions, such as iso-butyronitrile, acetone, 2-methyl-furan, or benzene. These compounds can be from the pyrolysis of cellulose and hemicellulose (Greenberg et al. 2006).

The emission ratio of total terpenoids was 61.200 $\mu\text{g}\cdot\text{g}^{-1}$ from heated needles in Table 2. The result is coincident with the value from Norway spruce sawdust during drying (Granström 2003). Granström (2003) reported that the emission ratios of monoter-

penes of drying sawdust were from $13 \mu\text{g}\cdot\text{g}^{-1}$ to $250 \mu\text{g}\cdot\text{g}^{-1}$ (oven-dry weight) depending on the temperature (140°C , 170°C or 200°C).

Table 2. Terpenoid emissions from heated needles (200°C , 15min) of *P. pumila* in mid-Sep., 2009. Values were means \pm s.e.m. ($n=3$).

Compounds	Emission ratio ($\mu\text{g}\cdot\text{g}^{-1}$)	Proportion of terpenoids (%)
Santene	1.017 ± 0.075	1.63
α -pinene	37.223 ± 2.136	59.60
Camphene	8.900 ± 0.092	14.25
β -pinene	7.452 ± 0.103	11.93
3-carene	1.801 ± 0.112	2.88
β -ocimene	0.474 ± 0.213	0.76
D-limonene	3.513 ± 0.142	5.63
β -phellandrene	0.607 ± 0.215	0.97
eucalyptol	0.213 ± 0.128	0.34
neo-allo-ocimene	1.262 ± 0.263	2.02
Total	61.200 ± 2.569	100.00

VOC emissions from heated twigs

Among 40 identified compounds from heated twigs, the most VOC were terpenoids, accounting for 92.40% of the total VOC (Fig. 4). The large amounts of compounds were illustrated in Fig. 5. The small amounts of other compounds were: (1) iso butyronitrile, (2) furan, (3) 5-hydroxy-2-pentanone, (4) ethylbenzene, (5) 4-carene, (6) 4-isopropyltoluene, (7) β -(Z)-ocimene, (8) (Z)-2-dodecene, (9) (-)-myrtenal, (10)tridecane, (11) (-)-verbenone, (12) 2,6,10-trimethyl-dodecane, (13) tetradecane.

Among 20 terpenoid compounds (Table 3), the main components were monoterpenes, such as α -pinene (35.72%), camphene (19.71%), β -pinene (15.34%), tricyclene (7.31%), D-limonene (6.72%), 3-carene (4.85%), neo-allo-ocimene (4.43%). There were also oxygen-monoterpenes ($\text{C}_{10}\text{H}_{14}\text{O}$), i.e., 2(10)-pinen-3-one, myrtenal, verbenone, but their amount were very small, accounting for 0.11%, 0.06% and 0.05% of the total terpenoids, respectively.

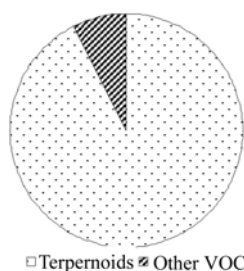


Fig. 4 Terpenoids and other VOC in the total emissions from heated twigs (200°C , 15 min) of *P. pumila* in mid-Sep., 2009

Terpenoids emitted from heated twigs are presented in Table 3. The total emission ratio was $217.060 \mu\text{g}\cdot\text{g}^{-1}$, which was 3.5 times to that of needles. The main reason for higher emission ratio from heated twigs is that twigs of *P. pumila* have more volatile oils than needles. In Fig. 6, it is shown that lots of oil drops on

Aluminum foil were under twigs after experiment, however, there was no oil drop on Aluminum foil under needles.

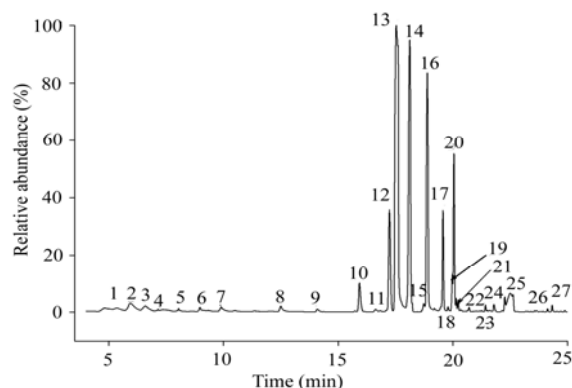


Fig. 5 Chromatographic profiles of VOC emissions from heated needles (200°C , 15 min) of *P. pumila* in mid-Sep., 2009

(1) pentane; (2) methylene chloride; (3) hexane; (4) 2-methyl-furan; (5) tri-chloro methane; (6) benzene; (7) 1-methoxy-2-propanol; (8) toluene; (9) hexanal; (10) sabinene; (11) fenchene; (12) tricyclene; (13) α -pinene; (14) camphene; (15) sabinene; (16) β -pinene; (17) 3-carene; (18) α -terpinene; (19) β -ocimene; (20) limonene; (21) β -phellandrene; (22) γ -terpinene; (23) terpinolene; (24) 1-methyl-4-(1-methylethenyl)-benzene; (25) neo-allo-ocimene; (26) 2(10)-pinen-3-one; (27) decanal.

Table 3. Terpenoid emissions from heated twigs (200°C , 15min) of *P. pumila* in mid-Sep., 2009. Values were means \pm s.e.m. ($n=3$).

Compounds	Emission ratio ($\mu\text{g}\cdot\text{g}^{-1}$)	Proportion of terpenoids (%)
santene	4.524 ± 0.321	2.08
fenchene	0.506 ± 0.106	0.23
tricyclene	15.866 ± 1.234	7.31
α -pinene	77.530 ± 2.003	35.72
camphene	42.788 ± 1.558	19.71
4-carene	0.334 ± 0.113	0.15
sabinene	1.179 ± 0.103	0.54
β -pinene	33.304 ± 1.224	15.34
3-carene	10.530 ± 1.002	4.85
α -terpinene	0.597 ± 0.105	0.27
(E)- β -ocimene	2.636 ± 0.605	1.21
D-limonene	14.580 ± 1.229	6.72
β -phellandrene	1.023 ± 0.356	0.47
β -(Z)-ocimene	0.328 ± 0.121	0.15
γ -terpinene	0.560 ± 0.142	0.26
terpinolene	0.687 ± 0.234	0.32
neo-allo-ocimene	9.613 ± 1.062	4.43
2(10)-pinen-3-one	0.247 ± 0.128	0.11
myrtenal	0.122 ± 0.091	0.06
verbenone	0.102 ± 0.086	0.05
Total	217.060 ± 2.567	100.00

The main components of VOC emissions from heated needles and twigs are similar, i.e., terpenoids, especially monoterpenes. The main components of monoterpenes are α -pinene, camphene, β -pinene, tricyclene, D-limonene, 3-carene, neo-allo-ocimene and santene (Table 2 and Table 3). The totals of these monoter-

penes account for 97.94% and 97.16% of the total terpenoids respectively. In addition, tricyclene was only detected in the VOC emissions from heated twigs (Table 3).

Though the main components are similar, there are many differences in components of VOC emissions from heated needles and heated twigs. Only 10 terpenoids were detected in VOC from heated needles, but 20 terpenoids were detected in VOC from heated twigs. The proportion of α -pinene in terpenoids is

59.60% and 35.72% of the total terpenoids, from heated needles and from heated twigs respectively. However, the terpenoid emission ratio from heated twigs is higher than that from needles. The reasons for these results are that the amount of volatile oils in twigs is higher than that in needles (Fig. 6). Consequently, some compounds can not be detected in VOC from heated needles because of small amount.



Fig. 6 Needles and twigs of *P. pumila* before and after experiment. The oil drops were shown in above circles for (a) air-dried needles; (b) heated needles (200 °C, 15min); (c) air-dried twigs; (d) heated twigs (200 °C, 15 min).

The terpenoid emissions from heated needles and twigs are very considerable. Zhao et al. (2004) reported that the terpenoid emissions rates from *Larix gmelini*, *P. koraiensis*, under standard conditions ($T=303\text{K}$, $\text{PAR}=1000\ \mu\text{mol}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$) in Helongjiang Province only were $0.05\ \mu\text{gC}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$, $0.30\ \mu\text{gC}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ respectively. The best interpretation for lower terpenoid emissions under natural conditions and higher terpenoid emissions under high temperature conditions is that terpenoids have a close relationship with temperature (Maleknia et al. 2009b). It can be inferred that a large amount of terpenoids will be emitted when fuels get heat from surrounding environment. More content of volatile oils is in tissues of fuels, more terpenoids will be emitted from heated fuels.

Conclusions

The present study focused on the VOC emissions from heated needles and twigs of *P. pumils*. Though there are differences in the components of VOC between needles and twigs, most of

VOC from heated needles and twigs are all monoterpenes. Terpenoid emissions from heated needles and twigs account for 72.93% and 92.40% of the total VOC respectively. Comparing with the terpenoid emissions under natural conditions, the terpenoid emission ratios under 200°C are very considerable, which are $61.200\ \mu\text{g}\cdot\text{g}^{-1}$ and $217.060\ \mu\text{g}\cdot\text{g}^{-1}$ from needles and twigs respectively. In actual fires, the large amounts of terpenoid emissions, especially the monoterpene emissions from heated fuels have strong effects on fires not only in the initial stage but also in fast propagation stage because these gases are very flammable with high calorific value. At the initial stage of fires, though the needles and twigs do not burn, the heat in surrounding environment stimulates a large number of terpenoids emitting to the air. These flammable gases can provide an advantage for flame combustion and fire spreading. At the fast propagation stage of fires, they are capable of causing violent combustion and creating crown fires. An uneven geographical area, such as a valley, can result in an accumulation of them. Consequently, when fires spread in such areas, the flame will propagate at unusual speed, and there will be a possible for eruptive fires and/or fires flash-

over to occur. As a consequence, the large amount of terpenoid emissions from heated fuels should be considered more carefully in fire suppression activities.

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